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Commissioner for Patents Washington, D.C. 20231

PCT/JP99/07006 ~ -filed December 14, 1999

Application of Masayuki YAMANA, Tsukasa AGA, Masahiro MIYAHARA,

Masaki FUKUMORI and Ryosuke HARA

AQUEOUS WATER- AND OIL-REPELLENT DISPERSION

Assignee: DAIKIN INDUSTRIES, LTD.

Our Ref: Q64814



The following documents and fees are submitted herewith in connection with the above application for the purpose of entering the National stage under 35 U.S.C. § 371 and in accordance with Chapter II of the Patent Cooperation Treaty:

☑ an English translation of the International Application.

☑ a Notification Concerning Submission or Transmittal of Priority Document.

☐ an English translation of Article 34 amendments (annexes to the IPER).

☑ an Information Disclosure Statement and Form PTO-1449 listing the ISR references.

The Declaration and Power of Attorney and Assignment will be submitted at a later date.

It is assumed that copies of the International Application, the International Search Report, the International Preliminary Examination Report, and any Articles 19 and 34 amendments as required by § 371(c) will be supplied directly by the International Bureau, but if further copies are needed, the undersigned can easily provide them upon request.

Assignment for published patent application is: DAIKIN INDUSTRIES, LTD..

The Government filing fee, after entry of the Article 34 Amendment, is calculated as follows:

Total claims	3 - 20	=	x \$18.00	=	\$.00
Independent claims	1 - 3	=	x \$80.00	=	\$.00
Base Fee		-	•		\$860.00
TOTAL FEE					\$860.00

A check for the statutory filing fee of \$860.00 is attached. You are also directed and authorized to charge or credit any difference or overpayment to Deposit Account No. 19-4880. The Commissioner is hereby authorized to charge any fees under 37 C.F.R. §§ 1.16, 1.17 and

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1.492 which may be required during the entire pendency of the application to Deposit Account No. 19-4880. A duplicate copy of this transmittal letter is attached.

Priority is claimed from December 22, 1998 based on Japanese Application No. 364298/1998.

Respectfully submitted,

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Date: June 21, 2001

Abraham J. Rosner Registration No. 33,276

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DESCRIPTION

AQUEOUS WATER- AND OIL-REPELLENT DISPERSION

5 FIELD OF THE INVENTION

The present invention relates to an aqueous water- and oil-repellent dispersion comprising a polymer containing a polymerizable compound having a perfluoroalkyl or perfluoroalkenyl group and an acrylate or methacrylate group, which can impart the water- and oil repellency to various fibers.

RELATED ARTS

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It is well known that a polymer of a polymerizable compound having a perfluoroalkyl or perfluoroalkenyl group and an acrylate or methacrylate group can be used as a water- and oil-repellent for a fibrous fabric. An aqueous dispersion which is prepared by dispersing the polymer in an aqueous medium by means of an emulsifier widely has the industrial utilization. However, the water- and oilrepellency fibrous fabric of a treated with usual conventional aqueous dispersions is not satisfactory for resistance, namely durability, to physical action such as friction. In addition, the conventional dispersions do not have excellent storage stability even if they have the durable water- and oil repellency.

SUMMARY OF THE INVENTION

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One of the objects of the present invention is to provide an aqueous water- and oil-repellent dispersion having durable water- and oil-repellency and excellent storage stability.

The present invention provides an aqueous water- and oil-repellent dispersion comprising:

- 10 (A) a homopolymer or copolymer comprising at least one polymerizable compound having a perfluoroalkyl or perfluoroalkenyl group and an acrylate or methacrylate group, or
 - a copolymer comprising said polymerizable compound and another compound copolymerizable therewith,
 - (B) an organic solvent which is tripropylene glycol, and
 - (C) a surfactant.

DETAILED EXPLANATION OF THE INVENTION

In the copolymer, which is the polymer (A), comprising the polymerizable compound having the perfluoroalkyl or perfluoroalkenyl group and the acrylate or methacrylate group and the another compound copolymerizable therewith, the former compound is in the amount of at least 25 % by weight, preferably at least 40 % by weight based on the

weight of the copolymer.

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Examples of the polymerizable compound having the perfluoroalkyl or perfluoroalkenyl group and the acrylate or methacrylate group include (meth)acrylate esters represented by the formulas:

$$R^{1}$$

$$Rf-SO_{2}-NR^{2}OCOCR^{3}=CH_{2}$$
(1)

$$Rf-(CH2)nOCOCR3=CH2 (2)$$

$$R_1$$
 Rf -CO-NR²OCOCR³=CH₂ (3)

$$Rf-O-Ar-CH_2OCOCR^3=CH_2$$
 (6)

wherein Rf is a perfluoroalkyl or perfluoroalkenyl group having 3 to 21 carbon atoms,

10 R^1 is a hydrogen atom or an alkyl group having 1 to 10 carbon atoms,

 R^2 is an alkylene group having 1 to 10 carbon atoms, R^3 is a hydrogen atom or a methyl group, Ar is an aryl group which optionally has a substituent group, and

5 n is an integer of 1 to 10.

Specific examples of the polymerizable compound include:

$$CF_{3}(CF_{2})_{7}(CH_{2})OCOCH=CH_{2},$$

$$CF_{3}(CF_{2})_{6}(CH_{2})OCOC(CH_{3})=CH_{2},$$

$$(CF_{3})_{2}CF(CF_{2})_{6}(CH_{2})_{2}OCOCH=CH_{2},$$

$$CF_{3}(CF_{2})_{7}(CH_{2})_{2}OCOC(CH_{3})=CH_{2},$$

$$CF_{3}(CF_{2})_{7}(CH_{2})_{2}OCOCH=CH_{2},$$

$$CF_{3}(CF_{2})_{7}SO_{2}N(CH_{3})(CH_{2})_{2}OCOCH=CH_{2},$$

$$CF_{3}(CF_{2})_{7}SO_{2}N(C_{2}H_{5})(CH_{2})_{2}OCOC(CH_{3})=CH_{2},$$

$$(CF_{3})_{2}CF(CF_{2})_{6}CH_{2}CH(OCOCH_{3})CH_{2}OCOC(CH_{3})=CH_{2},$$

$$(CF_{3})_{2}CF(CF_{2})_{6}CH_{2}CH(OH)CH_{2}OCOCH=CH_{2},$$

$$C_{9}F_{17}O-CH_{2}O-COCH=CH_{2}$$
and
$$C_{6}F_{11}-O-CH_{2}O-COC(CH_{3})=CH_{2}$$

The another copolymerizable compound are various.

Examples of the another copolymerizable compound include:

(1) acrylic acid and methacrylic acid, and methyl, ethyl,
butyl, isobutyl, t-butyl, propyl, 2-ethylhexyl, hexyl,

decyl, lauryl, stearyl, isobornyl, β -hydroxyethyl, glycidyl, phenyl, benzyl and 4-cyanophenyl esters thereof; (2) vinyl esters of fatty acids such as acetic acid, propionic acid, caprylic acid, lauric acid and stearic acid; (3) styrene compounds such as styrene, α -methylstyrene and methylstyrene; (4) vinyl halides and vinylidene compounds such as vinyl fluoride, vinyl chloride, vinyl bromide, vinylidene fluoride and vinylidene chloride; (5) fatty acid allyl esters such as allyl heptanoate, allyl caprylate and allyl caproate; (6) vinyl alkyl ketones such as vinyl methyl ketone and vinyl ethyl ketone; (7) acryl amides such as N-methylacrylamide and N-methylolmethacrylamide; and (8) dienes such as 2,3-dichloro-1,3-butadiene and isoprene.

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The organic solvent in the present invention is tripropylene glycol, H[OCH2CH(CH3)]3OH.

The amount of the organic solvent (B) may be from 5 to 200 parts by weight, for example from 10 to 100 parts by weight, particularly from 20 to 60 parts by weight, based on 100 parts of the polymer (A).

The surfactant (C) used for dispersing the polymer and the organic solvent may be a cationic emulsifier, an anionic emulsifier or a nonionic emulsifier. The surfactant is preferably the cationic emulsifier, the nonionic emulsifier or a mixture of both. In the case of the mixture, a preferable weight ratio of the cationic

emulsifier to the nonionic emulsifier is from 1:9 to 9:1. Specific examples of the cationic emulsifier include dodecyl trimethyl ammonium acetate, trimethyl tetradecyl ammonium chloride, hexadecyl trimethyl ammonium bromide, trimethyl octadecyl ammonium chloride, (dodecylmethyltrimethyl benzyl) ammonium chloride, benzyl dimethyl ammonium chloride, methyl dodecyl di(hydropolyoxyethylene) ammonium chloride, benzyl dodecyl di(hydropolyoxyethylene) ammonium chloride, benzyl dodecyl di(hydropolyoxyethylene) ammonium chloride and N-[2-(diethylamino)ethyl]oleamide hydrochloride. Specific examples of the nonionic emulsifier include a condensation product of ethylene oxide with hexyl phenol, iso-octyl phenol, hexadecanol, oleic acid, alkane($C_{12}-C_{16}$)thiol, monofatty acid (C_7-C_{19}) or alkyl $(C_{12}-C_{18})$ amine and the like.

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The amount of the surfactant (C) may be from 0.01 to 30 parts by weight, for example from 1 to 20 parts by weight, based on 100 parts by weight of the polymer (A).

The dispersion according to the present invention can be prepared by emulsion-polymerizing the polymerizable compound(s) in water accompanied by the organic solvent (B) in the presence of the surfactant to give an emulsion of the polymer (A). Water and/or the surfactant may be added to the emulsion of the polymer (A).

25 Examples of a suitable substrate, to which the

dispersion according to the present invention is applied, include a film, a fiber, a yarn, a woven fabric, a carpet, a filament made from a natural polymer material, a modified natural polymer material and a synthetic polymer material, and a product made from a fiber and a yarn. The substrate is preferably a textile which is in the form of a fiber, a yarn or a fabric.

The dispersion according to the present invention can be applied to the substrate preferably by coating, dipping, spraying, padding, roll coating, or combination of these procedures. For example, a padding bath having the bath solid content of 0.1 to 10 % by weight can be used. The substrate is padded in the bath, and then excess liquid is usually removed by a squeezing roll to give the dry pickup amount (the weight of dry polymer on the substrate) of from 0.01 to 10 % by weight based on the weight of the substrate. Then, the treated substrate is preferably heated at 100-200 °C.

20 PREFERRED EMBODIMENTS OF THE INVENTION

Examples and Comparative Examples are shown hereinafter to illustrate the present invention in detail.

Properties are determined as follows:

Water- and oil-repellency

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The polymer dispersion liquid is diluted with water to

give a treatment liquid having a solid content of 0.08 % by weight. A polyester fabric is immersed in the treatment liquid, squeezed with a mangle to give a wet pickup of 65%, dried at 100°C for two minutes, heated at 160°C for one hour, and then subjected to an evaluation of water- and oil-repellency.

The water-repellency is expressed by the water repellency No. (cf. the following Table 1) determined by the spray method according to JIS (Japanese Industrial Standard) L-1092.

The oil-repellency is determined by dropping several drops of a test solution shown in AATCC-TM118 (Table 2) on two positions of a surface of a test cloth and observing the penetration state of the drops after 30 seconds. The maximum point at which the test solution exhibits no penetration is expressed by the oil-repellency.

Table 1

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Water repellency	State		
No.			
5	No wet on the surface		
4	Slight wet on the surface		
3	Partial wet on the surface		
2	Wet on the surface		
1	Wet over the whole surface		

Table 2

Oil	Test solution	Surface
repel-		tension
lency		(dyne/cm,
		25°C)
8	n-Heptane	20.0
7	n-Octane	21.8
6	n-Decane	23.5
5	n-Dodecane	25.0
4	n-Tetradecane	26.7
3	n-Hexadecane	27.3
2	n-Hexadecane/Nujol mixture solution	29.6
	(35/65 by weight)	
1	Nujol	31.2
0	Inferior to 1	_

Storage stability

After the aqueous dispersion (solid content: 30 % by weight) is stored at 40°C for one month, the generation of precipitation is observed.

O: No precipitation

 \triangle : Slight precipitation

X: Much precipitation

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Example 1

100 g of $C_nF_{2n+1}CH_2CH_2OCOCH=CH_2$ (a mixture of compounds wherein n is 6, 8, 10, 12 and 14 (average of n is 8)), 50 g of stearyl acrylate, 2 g of N-methylol acrylamide, 200 g of pure water, 40 g of tripropylene glycol, 0.3 g of acetic acid, 4 g of alkyl trimethyl ammonium chloride and 10 g of polyoxyethylenealkylphenyl ether were charged into a 500 mL

flask and emulsified at 60°C for 15 minutes by means of ultrasonic wave with stirring. 0.75 g of azobisisobutylamidine dihydrochloride was added and the reaction was continued for 5 hours to give an aqueous dispersion of a polymer.

The water- and oil-repellency and the storage stability were evaluated. The results are shown in Table 3.

Example 2

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100 g of C_nF_{2n+1}CH₂CH₂OCOCH=CH₂ (a mixture of compounds wherein n is 6, 8, 10, 12 and 14 (average of n is 8)), 50 g of stearyl acrylate, 2 g of 3-chloro-2-hydroxypropyl methacrylate, 200 g of pure water, 30 g of tripropylene glycol, 0.3 g of acetic acid, 4 g of alkyl trimethyl ammonium chloride and 10 g of polyoxyethylenealkylphenyl ether were charged into a 500 mL flask and emulsified at 60°C for 15 minutes by means of ultrasonic wave with stirring. 0.75 g of azobisisobutylamidine dihydrochloride was added and the reaction was continued for 5 hours to give an aqueous dispersion of a polymer.

The water- and oil-repellency and the storage stability were evaluated. The results are shown in Table 3.

Example 3

25 100 g of $C_nF_{2n+1}CH_2CH_2OCOCH=CH_2$ (a mixture of compounds

wherein n is 6, 8, 10, 12 and 14 (average of n is 8)), 25 g of stearyl acrylate, 25 g of 2-ethylhexyl methacrylate, 200 g of pure water, 80 g of tripropylene glycol, 0.3 g of acetic acid, 4 g of alkyl trimethyl ammonium chloride and 10 g of polyoxyethylenealkyl ether were charged into a 500 mL flask and emulsified at 60°C for 15 minutes by means of ultrasonic wave with stirring. 0.75 g of azobisisobutyl-amidine dihydrochloride was added and the reaction was continued for 5 hours to give an aqueous dispersion of a polymer.

The water- and oil-repellency and the storage stability were evaluated. The results are shown in Table 3.

Example 4

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100 g of C_nF_{2n+1}CH₂CH₂OCOCH=CH₂ (a mixture of compounds wherein n is 6, 8, 10, 12 and 14 (average of n is 8)), 50 g of stearyl acrylate, 2 g of diacetone acrylamide, 200 g of pure water, 50 g of tripropylene glycol, 0.3 g of acetic acid, 4 g of alkyl trimethyl ammonium chloride and 10 g of polyoxyethylenealkylphenyl ether were charged into a 500 mL flask and emulsified at 60°C for 15 minutes by means of ultrasonic wave with stirring. 0.75 g of azobisisobutylamidine dihydrochloride was added and the reaction was continued for 5 hours to give an aqueous dispersion of a polymer.

The water- and oil-repellency and the storage stability were evaluated. The results are shown in Table 3.

Example 5

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100 g of $C_nF_{2n+1}CH_2CH_2OCOCH=CH_2$ (a mixture of compounds wherein n is 6, 8, 10, 12 and 14 (average of n is 8)), 25 g of stearyl acrylate, 25 g of 2-ethylhexyl methacrylate, 2 g of N-methylolacrylamide, 200 g of pure water, 60 g of tripropylene glycol, 0.3 g of acetic acid, 4 g of alkyl trimethyl ammonium chloride and 10 g of polyoxyethylenealkyl ether were charged into a 500 mL flask and emulsified at $60^{\circ}C$ for 15 minutes by means of ultrasonic wave with stirring. 0.75 g of azobisisobutylamidine dihydrochloride was added and the reaction was continued for 5 hours to give an aqueous dispersion of a polymer.

The water- and oil-repellency and the storage stability were evaluated. The results are shown in Table 3.

Comparative Example 1

The procedure of Example 1 was repeated except that 40 g of propylene glycol was used instead of 40 g of tripropylene glycol.

Comparative Example 2

The procedure of Example 1 was repeated except that 40

g of dipropylene glycol was used instead of 40 g of tripropylene glycol.

Comparative Example 3

The procedure of Example 2 was repeated except that 30 g of propylene glycol was used instead of 30 g of tripropylene glycol.

Comparative Example 4

The procedure of Example 2 was repeated except that 30 g of dipropylene glycol was used instead of 30 g of tripropylene glycol.

Comparative Example 5

The procedure of Example 4 was repeated except that 50 g of propylene glycol was used instead of 50 g of tripropylene glycol.

Table 3

		Water- and oil repellency					Storage
	Initi	al		Durabi			stabi-
			HL	-3	DC-3		lity
	Water-	Oil-	Water-	Water-	Water-	Water-	
	repel-	repel-	repel-	repel-	repel-	repel-	
	lency	lency	lency	lency	lency	lency	
Ex. 1	5	6	4	4	4	3	0
Ex. 2	5	6	4	4	4	3	0
Ex. 3	5	7	4	5	4	4	0
Ex. 4	5	6	4	4	4	3	0
Ex. 5	5	7	4	5	4	4	0
Com.	5	6	4	4	4	3	×
Ex. 1							
Com.	5	6	4	4	4	3	\triangle
Ex. 2							
Com.	5	6	4	4	4	3	×
Ex. 3							
Com.	5	6	4	4	4	3	×
Ex. 4							
Com. Ex. 5	5	6	4	4	4	3	×

Note) HL-3: After 3 times washing according to a JIS L-0217-103 method

DC-3: After 3 times dry cleaning according to a JIS I-1092-322 method

5 EFFECTS OF THE INVENTION

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Tripropylene glycol used in the present invention is excellent in no harm. The aqueous dispersion according to the present invention is effective for decreasing the environmental pollution and has durable water- and oil-repellency and excellent storage stability.

(amended under Article 34 PCT, UN 200 received on December 21, 2000 by IPEA)

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CLAIMS

- 1. (amended) An aqueous water- and oil-repellent
 dispersion comprising:
- 5 (A) a homopolymer or copolymer comprising at least one polymerizable compound having a perfluoroalkyl or perfluoroalkenyl group and an acrylate or methacrylate group, or
 - a copolymer comprising said polymerizable compound and another compound copolymerizable therewith,
 - (B) an organic solvent which is tripropylene glycol, and
 - (C) a surfactant,

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wherein the amount of the organic solvent (B) is from 5 to 200 parts by weight, based on 100 parts by weight of the polymer (A), and the polymer (A) is emulsion-polymerized in water accompanied by the organic solvent (B).

2. (deleted)

- 20 3. The dispersion according to claim 1, wherein the amount of the organic solvent (B) is from 10 to 100 parts by weight, based on 100 parts by weight of the polymer (A).
- 4. A textile, to which the dispersion according to claim 125 is applied.

ABSTRACT

An aqueous water— and oil-repellent dispersion containing: (A) a homopolymer or copolymer containing at least one polymerizable compound having a perfluoroalkyl or perfluoroalkenyl group and an acrylate or methacrylate group, or a copolymer containing said polymerizable compound and another compound copolymerizable therewith, (B) an organic solvent which is tripropylene glycol, and (C) a surfactant, has durable water— and oil-repellency and excellent storage stability.

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Declaration and Power of Attorney for Patent Application

特許出願宣言書及び委任状

Japanese Language Declaration

日本語宣言書

下記の氏名の発明者として、私は以下の通り宣言します。	As a below named inventor, I hereby declare that:		
私の住所、私書箱、国籍は下記の私の氏名の後に記載された通りです。	My residence, post office address and citizenship are as stated next to my name,		
下記の名称の発明に関して請求範囲に記載され、特許出願している発明内容について、私が最初かつ唯一の発明者(下記の氏名が一つの場合)もしくは最初かつ共同発明者であると(下記の名称が複数の場合)信じています。	I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled		
	AQUEOUS WATER- AND OIL-REPELLENT		
	DISPERSION /		
上 紀 発明 の 明 細 書 (下 記 の 横 で X 印 が つ い て い な い 場 合 は、本 書 に 添 付) は、	the specification of which is attached hereto unless the following box is checked:		
□月日に提出され、米国出願番号または特許協定 条約 国際出願番号をとし、	Was filed on <u>December 14, 1999</u> as Moited States Application Number PCT International Application Number		
(該当する場合) に訂正されました。	PCT/JP99/07006 and was amended on		
(December 21, 2000 (if applicable).		
私は、特許請求範囲を含む上記訂正後の明細書を検討し、内容を理解していることをここに表明します。	I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.		
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Japanese Language Declaration

(日本語宣言書)

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<u> </u>		Priority Not Claimed 優先権主張なし
Japan-	22/December/1998/	П
(Country) (国名)	(Day/Month/Year Filed) (出願年月日)	
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(Country) (国名)	(Day/Month/Year Filed) (出額年月日)	- -
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(Application No.)	(Filing Date)	(Status: Patented, Pending, Abandoned)	
(出願番号)	(出願日)	(現况:特許許可済、係属中、放棄済)	
(Application No.)	(Filing Date)	(Status: Patented, Pending, Abandoned)	_
(出願番号)	(出順日)	(現況:特許許可济、係属中、放棄済)	

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Japanese Language Declaration

(日本語宣言書)

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(第三以降の共同発明者についても同様に記載し、署名をするこ (Supply similar information and signature for third and subsequent joint inventors.)

Japanese Language Declaration (日本語宣言書)

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